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Evaluation of Monolithic Ceramics and Ceramic Thermal Barrier Coatings for Diesel Engine Applications

by Jeffrey J. Swab and Paul J. Huang

ARL-TR-2436

March 2001

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ARL-TR-2436

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Jeffrey J. Swab and Paul J. Huang

Weapons and Materials Research Directorate, ARL

Abstract

The Metals and Ceramics Research Branch (MCRB) of the Weapons and Materials Research Directorate is providing ceramic material characterization and evaluation to the Tank Automotive Research, Development, and Engineering Center (TARDEC) as a part of a project entitled "Fighting Vehicle Propulsion Technology Using Ceramic Materials." Through research and exploratory development of advanced ceramics and other technologies, the objective of this project is to improve the mobility of future Army fighting vehicles. The purpose is to demonstrate the capabilities of ceramics, other advanced materials, tribology, and combustion technologies to contribute to a higher power density and lower fuel consumption diesel engine.

Four commercial silicon nitrides were purchased and characterized for potential application as piston crowns, valves, and valve seats. A sodium-zirconium-phosphate (NZP) family of materials was examined for exhaust port applications, and several thermal barrier coating (TBC) materials were examined as possible additions to metallic piston crowns, rings, and cylinder liners. This report contains the results of this preliminary characterization and evaluation.

Acknowledgments

The authors would like to acknowledge the financial support of the Propulsion Systems Division of the U.S. Army Tank-Automotive Research, Development, and Engineering Center (TARDEC) provided by Dr. Walter Bryzik, Ernest Schwarz, and Eugene Danielson. The authors would also like to acknowledge the following people who assisted in this effort: Matthew Motyka for his efforts in the mechanical property testing of these materials, Dr. Parimal Patel for performing the thermal expansion testing, and Anand Kulnari at State University of New York (SUNY) for helping with the thermal conductivity tests.

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1. Introduction

The oil embargo of the 1970's spurred the need to improve the performance of engines to reduce the dependence on fossil fuel. Modern high performance ceramics with unique properties (high temperature strength, strength retention after time at high temperatures, and oxidation/corrosion resistance) were identified as materials that have the potential to increase engine performance and reduce fuel consumption. Since that time, significant efforts have been made by the materials and engine development communities to take advantage of advanced ceramic materials to increase engine performance and achieve this goal. Recently, environmental concerns have also contributed to the push to increase the applications of ceramic materials in engines. Although the benefits envisioned almost thirty years ago have yet to be fully realized, tremendous potential for ceramic materials to enhance engine performance still exists.

A variety of ceramic materials are being considered for applications in advanced engines. Monolithic ceramic materials (silicon nitride is the leading candidate), which are under consideration as structural components to replace metallic piston crowns, valves, and valve seats, have better corrosion resistance (ability to tolerate low-grade fuels), higher strength at elevated temperature, and higher hardness (better wear performance) than most metals. Other monolithic ceramics, specifically tailored with low thermal conductivity and thermal expansion, are being considered as liners for exhaust ports. Using a material with a low thermal conductivity in the exhaust port (the transfer of thermal energy to the cylinder head) can reduce the thermal energy loss at the cylinder head and lead to a smaller cooling system. Additional savings can also be gained in a turbo charging system by using the retained thermal energy to increase engine output power. Another class of ceramic materials with low thermal conductivity are ceramic thermal barrier coatings (TBCs). Ceramic TBCs are not designed as structural components, but instead are used to reduce the operating temperature a metallic component experiences, which extends the component life. It is anticipated that incorporating ceramic materials into diesel engines will reduce the in-cylinder heat rejection by almost 40%, improve fuel economy by approximately 6%, and cut particulate emission by up to 50%. Other benefits may include multifuel capability,

improved power output and combustion efficiency, decreases in engine size and weight, reduced noise levels, and reduced maintenance costs [1, 2].

The Metals and Ceramics Research Branch (MCRB) of the Weapons and Materials Research Directorate is providing ceramic material characterization and evaluation to the Tank and Automotive Research Development and Engineering Center (TARDEC), as part of an international program to examine advanced materials for diesel engines. Through research and exploratory development of advanced ceramics and other technologies, the objective is to improve the mobility of future Army fighting vehicles. The purpose is to demonstrate the capabilities of ceramics, other advanced materials, tribology, and combustion technologies to contribute to a diesel engine with a higher power density and lower fuel consumption.

The objective of this project will be accomplished through a series of five cooperative tasks: (1) a preliminary analysis of combustion phenomena, (2) an analysis of engine parameters, (3) ceramic material testing and evaluation, (4) the design, fabrication, and optimization of a test bed integrated single cylinder engine (TI-SCE), and (5) final testing and evaluation of the TI-SCE. The United States and its international partner* will be collaborating with major engine manufacturers in their respective countries to develop and test the TI-SCE. The main engine contractor for the DOD will be the Detroit Diesel Corporation, while the main engine contractors of the international partner are also leading diesel engine manufacturers.

As part of Task 3, MCRB is evaluating the thermal and mechanical properties of monolithic ceramics and thermal barrier-coating materials. The information generated will be incorporated into the development of the TI-SCE. A reciprocal agreement to exchange specimens and data with our international partner is part of the project. Ceramic materials will be examined to use in the following engine components: piston crowns, rings, cylinder heads and liners, valves, valve seats, and intake exhaust ports.

* The identity of the international partner is restricted to a need-to-know basis.

This report covers the initial screening and characterization of monolithic ceramic materials and ceramic thermal barrier-coating materials that have potential applications in the combustion area of advanced diesel engines. Additional testing is presently underway on the "best" materials and will be reported in the future. All materials evaluated were domestically produced and most are commercially available. The report is divided into three sections: Silicon Nitrides, Low Thermal Expansion/Low Thermal Conductivity Ceramics, and Ceramic TBCs. In each section, the properties measured and analyzed will be discussed and (if possible) compared to the values provided by the manufacturers.

2. Silicon Nitride

2.1 Background. Four silicon nitride (Si_3N_4) materials were examined for potential diesel engine applications such as piston crowns, valves, and valve seats (see Table 1). Recent studies in Japan [3] and Germany [4] using silicon nitride valves in automotive applications have reported great success. Over 500,000 km of road testing has been completed without any problems in the Japanese study, while the German effort produced 30,000 high-quality valves over a three-month period for field testing by Mercedes-Benz.

Table 1. Silicon Nitrides Evaluated

Manufacturer	Material Code	Billet Size ^a	Processing Technique
Allied Signal, Inc.	GS-44CL	50 ϕ \times 50 t	CIP/Sinter
Ceradyne, Inc.	Ceralloy 147-31N	100 \times 100 \times 9	Reaction Bonded
Cercom, Inc.	CIW15	150 \times 150 \times 8	Hot Pressed w/15 v/o SiC whiskers
St. Gobain Industrial Ceramics	NT551	57 \times 57 \times 13	Sinter/HIP

^aAll dimensions are nominal and in millimeters.

Typically, the material manufacturers produce a “family” of Si_3N_4 materials with each member of the family tailored to a specific application. After a discussion with each of the manufacturers, the GS-44CL, NT551, and Ceralloy 147-31N silicon nitrides listed in Table 1 were selected as the test materials. Each Si_3N_4 may be considered in-situ toughened and was developed with engine applications in mind. During processing, steps were taken to ensure the elongation of some grains to provide a duplex microstructure which could enhance some of the mechanical properties. (The CIW15 silicon nitride is not an in-situ toughened material. It is however a toughened silicon nitride, as it contains approximately 15 volume-percent SiC whiskers which have been added to enhance toughness. Although not designed for engine applications, this material was included to compare with the in-situ toughened silicon nitrides.) All materials were produced in 1997 and purchased in large billet sizes that were subsequently machined to the appropriate test specimen geometries. Only one billet was purchased of the CIW15, while multiple billets were needed from the other three silicon nitrides to obtain sufficient specimens for the entire test matrix.

Rectangular specimens with nominal dimensions of 3 mm x 4 mm x 50 mm were machined for all tests, except for determining thermal expansion and conductivity tests. All of the rectangular specimens were machined according to guidelines in ASTM C1161 [5]. For thermal expansion measurements, the specimens were 3 mm x 3 mm x 4 mm, and for thermal conductivity, a disk measuring 12.5 mm in diameter by 1 mm thick was used.

2.2 Test Results. The physical and mechanical properties determined in this study are listed in Table 2. Many of the properties listed in Table 2 have been determined elsewhere over the past 6–10 years for these same silicon nitrides [6–11]. However, it is virtually impossible to compare these sets of data without knowing the vintage of the material. Manufacturers are continually optimizing the composition and processing methods in order to make a better product and/or tailor the product to a specific application. Although most of these modifications are subtle, they can have a significant impact on a property or properties, which makes comparing different vintages of the same material a daunting (if not impossible) task. In this report, product data supplied by the manufacturers will be available for comparison. The manufacturers’ data is

Table 2. Properties of the Silicon Nitrides Evaluated

Product Data	GS-44CL		NT 551		Ceralloy 147-31N		CIW15	
	Company	ARL	Company	ARL	Company	ARL	Company	ARL
Density (g/cc)	3.2	3.21	3.29	3.28	3.3	3.20	3.27	3.25
Elastic Modulus (GPa)	310	356	310	347	320	305	335	380
Flexure Strength (MPa)	759	805 ⁱ	890-970	955 ⁱ	800	798 ⁱ	800	770 ⁱ
Weibull Modulus	20-35	30	NA	15	15-20	16	17	16
Toughness (MPa√m)	8.25 ^a	7.92 ± 0.19	6.5-6.9 ^c	6.31 ± 0.16	6-7.5 ^e	5.57 ± 0.22	10.2 ^g	6.03 ± 0.28
Hardness HV ₁ (GPa)	14.3 ^b	14.0	13.4 ^d	14.3	15.2 ^f	15.0	15.9 ^h	18.4
RT Thermal Conductivity (W/m K)	35	28	NA	22	35	23	NA	18
CTE RT-1000 °C (×10 ⁻⁶ /°C)	3.4	3.7	NA	4.3	3.5	3.3	3.3	4.4

Note: NA - not available.

^a Short beam chevron notch.

^b 10-kg load.

^c Indentation strength method.

^d Indentation load unknown.

^e Indentation - crack length with 5-kg vickers indent.

^f 5-kg load.

^g Single-edge notched beam.

^h Knoop hardness - 1-kg load.

ⁱ Characteristic flexure strength of the bend bar.

typical for the material they produced, but it may not be representative of the vintages tested in this study.

2.2.1 Density. Density was measured at room temperature using the Archimedes water immersion method with distilled water. A minimum of 10 specimens were used to determine the average density of each Si_3N_4 . When specimens were obtained from multiple billets, the density was determined on specimens randomly selected from all billets. This was done to check for possible billet-to-billet variability. The densities measured in this study are in agreement with the respective company data (3.20 g/cm^3 to 3.28 g/cm^3). No billet-to-billet variability was detected in the density data for any of these materials.

2.2.2 Elastic Modulus. Elastic modulus was obtained on a minimum of five specimens of each Si_3N_4 using the procedures in ASTM C1259 [12]. In this test method, the fundamental flexural resonant frequency is measured when a rectangular specimen is mechanically excited by a single elastic strike. A transducer senses the resultant mechanical vibration and transforms it into an electrical signal. The fundamental frequency is isolated, and this value, along with the specimen dimensions and mass, are used to calculate the dynamic elastic modulus. For all but the Ceralloy 147-31N, the elastic modulus obtained is considerably higher than the manufacturer's values. The reason for this discrepancy is unknown and additional analysis is needed.

2.2.3 Composition.* Semiquantitative energy dispersive x-ray fluorescence (XRF) analysis was used to determine the elemental composition of each Si_3N_4 . The analysis was completed on a fine powder under a vacuum of approximately 300 mTorr. Spectra were collected with a thin window solid state $\text{Si}(\text{Li})$ detector system using tungsten white radiation for excitation. Elements present in each material were determined from the spectra. This method is not capable of detecting Al ($n = 13$) or elements with lower atomic numbers; thus, the results in Table 3 may not reflect the complete elemental composition of each material.

*XRF analysis completed by Lamda Research Inc., Cincinnati, OH, under contract DAAD17-99-P-0338.

Table 3. Elemental Analysis of Si₃N₄

Si ₃ N ₄ Material→ Element (weight-percent)↓	GS-44CL	NT 551	Ceralloy 147-31N	CIW15
Si	92	82	82	88
Y	7	6	7	11
Yb	<1	<1	<1	<1
Ca	<1	—	—	<1
Fe	<1	—	—	<1
Co	<1	<1	<1	<1
Nd	—	11	11	—
La	—	<1	<1	—

Note: The dash (—) denotes an element not detected.

The presence of yttrium in each of these silicon nitrides is expected since it was found that Y₂O₃ is an excellent densification [13] aid for silicon nitride, and that the resultant Si₃N₄ exhibited excellent properties at room and elevated temperatures. In recent years, research has focused on other rare earth oxides (especially Nd₂O₃, La₂O₃ and CeO₂) and rare earth oxide in combination with Y₂O₃ to reduce the cost of the densification aid without compromising the properties [14–16].

2.2.4 Thermal Conductivity. The average room temperature thermal conductivity was determined using a laser flash method in accordance with JIS R1611 [17] on five specimens from each silicon nitride. Uniformly thick specimens were coated with a thin layer of graphite to minimize reflection of the pulse. The rise in temperature of the specimen as it is subjected to the laser pulse is obtained to determine the thermal diffusivity of the specimen. The thermal diffusivity, specimen density, and thickness are used to calculate the thermal conductivity. Values obtained in this study are consistently lower when compared to the manufacturer's values. This may be due to the use of different measurement techniques.

2.2.5 Thermal Expansion. The coefficient of thermal expansion from room temperature to 1000 °C was determined using a single-rod dilatometer. Specimens were heated at 100° C/hr,

and the CTE value was determined from the linear regression of the data. The resulting values listed in Table 2 are in good agreement with the manufacturer's values.

2.2.6 Mechanical Strength. The flexure strength was measured in accordance with ASTM C1161 using a standard 20 mm × 40 mm fully articulating four-point flexure fixture and a cross-head speed of 0.5 mm/min. A minimum of 30 longitudinally-machined specimens of each Si_3N_4 were fractured to obtain the average flexural strength. A two-parameter Weibull plot (using uncensored data) was then generated following the procedure in ASTM C1239 [18] to determine the characteristic flexure strength and unbiased Weibull modulus using maximum likelihood estimations. The characteristic flexural strength and Weibull modulus of each silicon nitride were in excellent agreement with the values reported by the manufacturers. Figure 1 contains the Weibull plots for each silicon nitride. These Weibull plots indicate that there is a single flaw population limiting the strength of each silicon nitride.

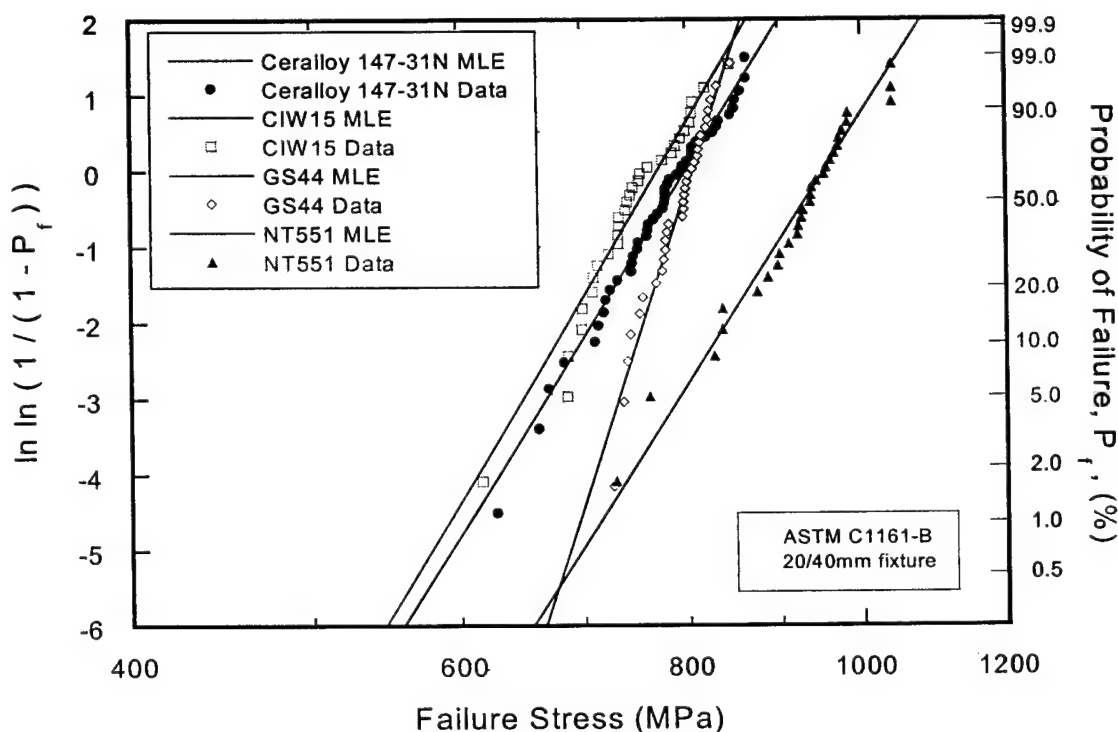


Figure 1. Two-Parameter Weibull Plot.

In order to check for possible billet-to-billet variability in cases where multiple billets were needed to obtain the requisite number of specimens, the 30 specimens were randomly and equally selected from each billet. A simple student t-test was used to determine if any differences existed in strength between billets. Such a difference was only observed in the Ceralloy 147-31N silicon nitride.

2.2.7 Fracture Analysis. The fracture surfaces from all 30 specimens of each silicon nitride were optically examined using a low magnification microscope. Selected fracture surfaces were examined in more detail using an SEM (according to ASTM C1322 [19]) to determine the strength-limiting flaw(s) in each material. Figures 2 (a–d) are SEM photographs that represent the dominate strength-limiting flaw type in each silicon nitride.

The dominant flaw in GS-44CL was a volume-distributed pore (see Figure 2[a]), while the strength of CIW15 was limited by a volume-distributed agglomerate of SiC whiskers, as in Figure 2(b). The fracture surfaces of the NT551 and Ceralloy 147-31N (Figures 2[c] and 2[d]) revealed that the strength of both was limited by surface flaws created during machining. Previous work reported [10] that under very similar testing conditions, machining-induced damage limited the strength of NT551. In the same study, NT551 exhibited a color inhomogeneity resembling a “snowflake” on the specimen surface when examined optically. The inhomogeneity was linked to the secondary phase in the material. Elemental mapping showed that the snowflake regions were comprised of SiO_2 , while the nonsnowflake regions contained Al_2O_3 , Y_2O_3 , and Nd_2O_3 . It was concluded that the inhomogeneity was the result of a separation of this secondary phase or that two different secondary phases are present as a result of the processing. The vintage of NT551 examined in this research also exhibited the same inhomogeneity.

2.2.8 Hardness. Vickers hardness (HV_1) was determined through the indentation of polished sections using a 1-kg indentation load and the procedures outlined in ASTM C1327 [20]. Five valid indentations were made on each specimen, and the average hardness was reported. In cases where multiple billets were used, the hardness from each billet was

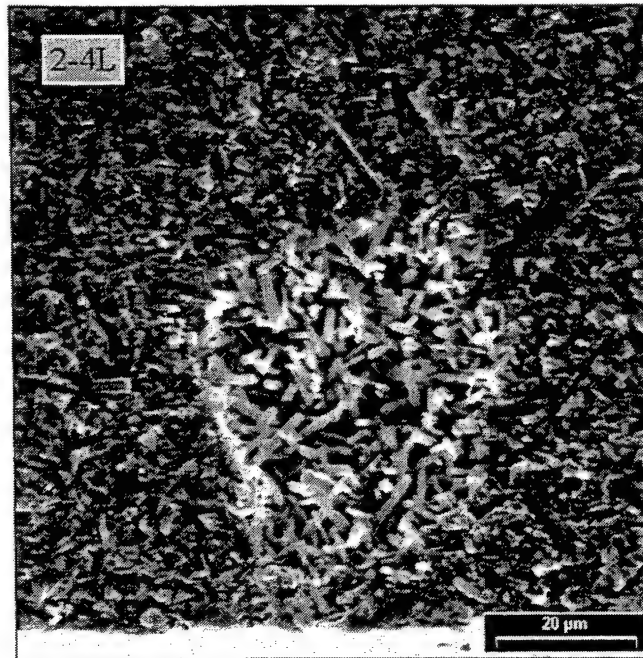


Figure 2(a). Strength-Limiting Flaw in GS-44CL Is a Pore (P^V , Surface, $\approx 30 \mu\text{m}$).

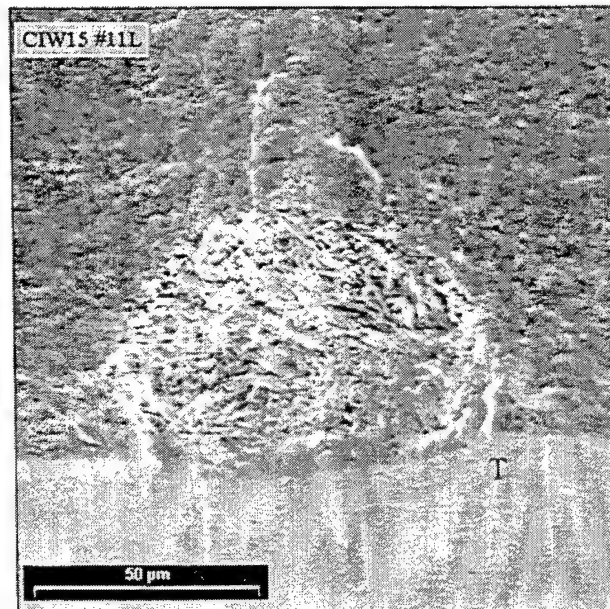


Figure 2(b). Strength-Limiting Flaw in CIW15 Is an Agglomerate of SiC Whiskers (A^V , Surface, $\approx 80 \mu\text{m}$). "T" Denotes the Specimen Tensile Surface.

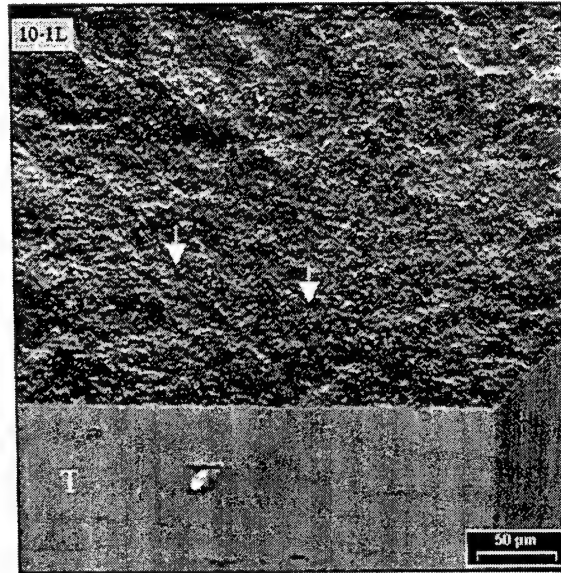


Figure 2(c). Strength-Limiting Flaw in NT551 Is Machining Damage (MD^S , Surface, $\approx 75 \mu m$). "T" Denotes the Specimen Tensile Surface, and the Arrows Indicate the Subsurface Semielliptical Cracks Created During Machining.

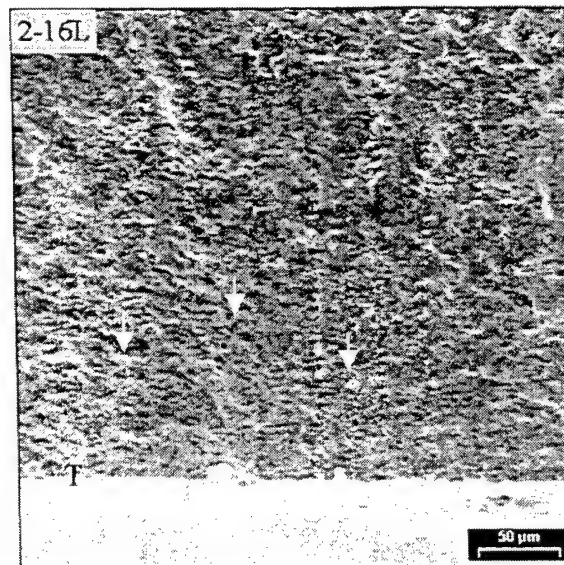


Figure 2(d). Strength-Limiting Flaw in Ceralloy 147-31N Is Machining Damage (MD^S , Surface, $\approx 60 \mu m \times 200 \mu m$). "T" Denotes the Specimen Tensile Surface, and the Arrows Indicate the Subsurface Semielliptical Crack Created During Machining.

determined. The hardness was consistent throughout the various billets in all materials. The HV_1 values of the GS-44CL, NT551, and Ceralloy 147-31N are in fairly good agreement with the manufacturer's values, even though these values were obtained with higher indentation loads. A direct comparison is impossible for CIW15 since the company reports Knoop hardness.

2.2.9 Fracture Toughness. The room temperature Mode I fracture toughness (K_{Ic}) was determined with a minimum of five specimens from each material using the single-edge precracked beam (SEPB) technique outlined in ASTM C1421 [21]. With its long diagonal perpendicular to the long axis of the specimen, a single Knoop indentation was placed in the middle of one 3-mm-wide face of each specimen using a 10-kg indentation load. A precrack was subsequently popped in using a compression anvil with a 6-mm bridge span. Specimens were then fractured in four-point flexure using inner and outer spans of 20 and 40 mm, respectively, and the length of the popped-in crack measured. The K_{Ic} measured in this study was consistently below that reported by the manufacturers. This is not surprising since the manufacturers use test methods which typically yield higher values of K_{Ic} . There were no observed billet-to-billet variations in toughness for any of these silicon nitrides.

2.3 Summary. The GS-44CL silicon nitride was selected to exchange with our international partner because it had a combination of highest fracture toughness, excellent strength, a high Weibull modulus, and because there was no billet-to-billet variability in the properties.

3. Low Thermal Expansion/Low Thermal Conductivity Ceramics

3.1 Background. A sodium-zirconium-phosphate (NZP) family of materials with low thermal expansion, low thermal conductivity, and high thermal shock resistance is produced by Low Thermal Expansion Ceramics, Co. (LoTEC). A 70% dense (low density) and an 80% dense (medium density) version of this material was purchased and evaluated for exhaust port applications.

3.2 Test Results. Specimens (the same dimensions as those used to determine the properties of the silicon nitrides) were machined from large billets nominally 200 mm × 200 mm × 13 mm in size. Unless noted, the testing procedures for property determination were the same as those previously used for the silicon nitrides. Table 4 is a summary of the material properties.

Table 4. Properties of the NZP Low Thermal Expansion Materials

Product Data	Low Density (70%)		Medium Density (80%)	
	Company	ARL	Company	ARL
Density (g/cc)	2.3–2.65	2.43	2.65–3.0	2.78
Elastic Modulus (GPa) ^a	50	40	50	63
Mean Flexure Strength (MPa) ^a	50	49	50	71
Weibull Modulus	NA	10	NA	10
Toughness (MPa√m) ^a	1.5–2	—	1.5–2	—
Hardness HV ₁ (GPa)	NA	0.97	NA	2.5
RT Thermal Conductivity (W/m °K)	≈1	0.48	≈1	0.83
CTE RT-1000°C (×10 ⁻⁶ /°C)	1	2.68	1	1.93

^aThe strength, fracture toughness, and elastic modulus data provided by the company are for a 90% dense material.

3.2.1 Density. Attempts to obtain a valid density for these porous ceramics using the Archimedes water immersion method were unsuccessful, as the resultant values were typically higher than the manufacturer's values. Simply immersing the specimen in distilled water was insufficient to fill the pores and provide an accurate saturated weight. A true saturated weight was achieved by placing the specimens in a desiccator, pulling a vacuum, and back filling with water. The densities determined by this modified method are in excellent agreement with the respective manufacturer's data for both versions of the material.

3.2.2 Elastic Modulus. Elastic modulus values are in excellent agreement with the manufacturer's values.

3.2.3 Composition. Table 5 summarizes the elemental content of both NZP materials. As expected, there is no difference in the elemental composition of the two versions of this material.

Table 5. Elemental Composition of NZP Materials

Element (weight-percent)	Material	
	Medium Density (80%)	Low Density (70%)
P	28	27
Sr	<1	<1
Zr	56	57
Ba	15	14
Fe	<1	<1
Hf	<1	1
Ni	—	<1

Note: The dash (—) denotes an element not detected.

3.2.4 Thermal Conductivity. Both of the thermal conductivity values reported are below the company value. The low-density NZP has a thermal conductivity of about one half of the medium-density NZP, due to the higher content of porosity in the former material.

3.2.5 Thermal Expansion. The thermal expansion values obtained in this study are higher than the manufacturer's values. This may be caused by differences in the test technique, the heating rates, or both.

3.2.6 Mechanical Strength. As expected, the flexural strength was very low for both versions because of the porous nature of the material. The average strength of each material is in excellent agreement with the values reported by the manufacturer. No fracture analysis was conducted due to the high porosity content and the difficulties associated with fractography of such materials.

3.2.7 Hardness. Hardness measurements were complicated by the porous nature of these NZPs—it took many indents to obtain five valid measurements. Since these are porous materials, it is not surprising that the hardness (HV_1) of both is relatively low.

3.2.8 Fracture Toughness. Due to the coarse-grained microstructure and the significant amount of porosity in both versions, valid fracture toughness values could not be obtained using the SEPB method. No other method was tried.

3.3 Summary. Both versions of the NZP material appear to have the necessary properties for consideration as exhaust port materials. The medium-density NZP was selected to exchange with our international partner because the material was readily available at the time of the exchange.

4. Ceramic Thermal Barrier Coatings (TBCs)

4.1 Background. Ceramic TBCs were examined for potential diesel applications such as coatings for piston crowns, rings, cylinder head and liner valves, valve seats, and intake exhaust ports. The TBC materials evaluated are listed in Table 6.

Table 6. Ceramic TBC Materials Evaluated

Manufacturer	Material	Bond Coat	Comments
Heany Industries	CeO ₂ -PSZ ^a	NiCrAlY	—
Heany Industries	CeO ₂ -PSZ (sealed)	NiCrAlY	Sol-gel sealed top layer
Heany Industries	Mullite	NiCrAlY	—
SUNY ^b at Stony Brook	8% Y ₂ O ₃ -PSZ	Ni-4.5Al	—
Praxair Surface Technologies	Y ₂ O ₃ -PSZ (LZ16)	CoNiCrAlY	—

^aPSZ - Partially stabilized zirconia.

^bSUNY - State University of New York.

The ceramic TBC materials evaluated were selected based on the attempt to push the current technology of TBC, and selection from a current commercial-grade coating as the baseline material. The three coatings selected from Heany Industries were selected to push the technology. The coating from the Thermal Spray Laboratory, SUNY at Stony Brook, was selected to see the traditional TBC with a novel bond coat on 4140 steel substrates. The coating from Praxair Surface Industries was selected as the baseline TBC. All materials were produced

using a standard atmospheric plasma spray technique, some were sprayed as coating only on removable substrate, and some were sprayed with a bond coat on 4140 steel substrates (as indicated in the test method section).

4.2 Test Results. The physical and mechanical properties of these coatings, as determined in this study, are listed in Tables 7 and 8. These property values vary from what has been previously reported in literature for similar types of TBCs. This is most likely due to differences in coating deposition parameters and different bond coats.

Table 7. Screening Data for Ceramic TBC Materials

Materials	Density (g/cc)	Hardness (HK ₁) (GPa)	Thermal Conductivity (W/m K)	Thermal Shock	Average CTE ($\times 10^{-6}/^{\circ}\text{C}$)
Ce-PSZ	5.22	2.1 ± 0.4	0.49	fail	13.4
Ce-PSZ (sealed)	5.54	3.2 ± 0.3	0.65	pass	11.7
Mullite	2.76	3.4 ± 0.2	0.78	fail	6.6
8% Y-PSZ	5.58	2.9 ± 0.1	0.83	fail	11.7
LZ16	5.46	5.3 ± 0.4	1.52	pass	N/A

Table 8. Adhesion Strength of Ceramic TBCs

Materials	No. of Specimens Tested	Average Adhesion Strength (MPa)
Ce-PSZ ^a	5	3.2
Ce-PSZ ^a	5	4.1
Ce-PSZ	4	7.5
LZ-16	4	2.7

^aTBC coating only, no bond coat used.

4.2.1 Density. The density was measured by the Archimedes method using an inert gas pycnometer at room temperature with 10-mm diameter TBC-only specimens. The average density was calculated from a minimum of 10 runs from three specimens. The measured density appears to agree with published results found in literature.

4.2.2 Hardness. The hardness test was measured in accordance to ASTM C1326 [22] using a Knoop microhardness indenter at room temperature with a 1-kg load and minimum of five valid indents to generate the standard deviation. The specimen was a polished cross-section of a 10-mm-diameter TBC with a bond coat on 4140 steel substrate.

4.2.3 Thermal Conductivity. The thermal conductivity test was in accordance to JIS R1611, as described previously, at room temperature on five, 10-mm-diameter TBC-only specimens.

4.2.4 Thermal Shock. The thermal shock testing was conducted by placing the 10-mm diameter TBC with a bond coat on 4140 steel substrate in a furnace at 800 °C for 30 min in air, and then rapidly quenching it in water at room temperature. This sequence constituted one cycle, and each specimen was subjected to only one cycle. Three specimens of each material were examined to see if delamination or cracking occurred at the interfaces. Each specimen was then classified as “pass” or “fail” based on whether delamination occurred. Visual examination indicated that delamination occurred at the bond coat/substrate interface, but an additional examination with the scanning electron microscope should be conducted to verify whether delamination occurred at the bond coat/substrate interface, or at the bond coat/TBC interface.

4.2.5 Thermal Expansion. The coefficient of thermal expansion (CTE) was measured from room temperature to 800 °C using a dilatometer. The test procedure was in accordance to ASTM E228-95 [23]. The CTE measurement was conducted through the coating thickness (parallel to the spray direction) of the coating. Future work will be conducted to measure the CTE in the direction normal to the spray direction.

4.2.6 Adhesion Strength. The adhesion strength was measured according to ASTM C633 [24]. This method is used to determine the adhesive strength of a coating to a substrate when subjected to a tensile stress field normal to the surface. In this study, testing consisted of coating one flat face of 1-inch-diameter cylindrical steel substrate with the TBC/bond coat system, and then bonding this coating to the face of an uncoated steel substrate with a two-part, room temperature curing epoxy. The entire assembly was then subjected to a tensile load normal to

the plane of the TBC, at room temperature, until failure. Testing was conducted on the Ce-PSZ and LZ-16 coatings, and the results are listed in Table 8. The Ce-PSZ materials were tested with and without bond coat (BC), and the LZ-16 material was tested with bond coat. As expected, the adhesion strength was significantly better with BC, but still lower than typically reported strengths for TBCs. This discrepancy is probably due to the much slower cross-head speed, which was set to 0.127 mm/min. A literature review shows that the cross-head speed is typically 1.27 mm/min. Future work will investigate the effect of cross-head speed on the adhesion strength.

4.3 Summary. The Ce-PSZ and Ce-PSZ sealed materials were selected as the “best” materials to exchange with our international partner since they had the lowest thermal conductivity and met the need to push ahead the current thermal barrier coating technology. Upon further evaluation, TBCs may not be viable candidates for certain engine components due to poor wear resistance and processing-related issues.

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Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project(0704-0188), Washington, DC 20503.				
1. AGENCY USE ONLY (Leave blank)	2. REPORT DATE March 2001	3. REPORT TYPE AND DATES COVERED Final, Oct 96 - Sep 99		
4. TITLE AND SUBTITLE Evaluation of Monolithic Ceramics and Ceramic Thermal Barrier Coatings for Diesel Engine Applications		5. FUNDING NUMBERS Oct 96-Sep 97—785K31 Oct 97-Sep 98—88M439 Oct 98-Sep 99—98M431		
6. AUTHOR(S) Jeffrey J. Swab and Paul J. Huang				
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) U.S. Army Research Laboratory ATTN: AMSRL-WM-MC Aberdeen Proving Ground, MD 21005-5069		8. PERFORMING ORGANIZATION REPORT NUMBER ARL-TR-2436		
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) TARDEC Propulsion System Division, Engine Research Function, AMSTA-TR-R, MS 121 Warren, MI 48397-5000		10. SPONSORING/MONITORING AGENCY REPORT NUMBER		
11. SUPPLEMENTARY NOTES				
12a. DISTRIBUTION/AVAILABILITY STATEMENT Approved for public release; distribution is unlimited.			12b. DISTRIBUTION CODE	
13. ABSTRACT (Maximum 200 words) The Metals and Ceramics Research Branch (MCRB) of the Weapons and Materials Research Directorate is providing ceramic material characterization and evaluation to the Tank Automotive Research, Development, and Engineering Center (TARDEC) as a part of a project entitled "Fighting Vehicle Propulsion Technology Using Ceramic Materials." Through research and exploratory development of advanced ceramics and other technologies, the objective of this project is to improve the mobility of future Army fighting vehicles. The purpose is to demonstrate the capabilities of ceramics, other advanced materials, tribology, and combustion technologies to contribute to a higher power density and lower fuel consumption diesel engine. Four commercial silicon nitrides were purchased and characterized for potential application as piston crowns, valves, and valve seats. A sodium-zirconium-phosphate (NZP) family of materials was examined for exhaust port applications, and several thermal barrier coating (TBC) materials were examined as possible additions to metallic piston crowns, rings, and cylinder liners. This report contains the results of this preliminary characterization and evaluation.				
14. SUBJECT TERMS ceramics, silicon nitride, thermal barrier coatings, diesel engines, partially stabilized zirconia			15. NUMBER OF PAGES 31	
			16. PRICE CODE	
17. SECURITY CLASSIFICATION OF REPORT UNCLASSIFIED	18. SECURITY CLASSIFICATION OF THIS PAGE UNCLASSIFIED	19. SECURITY CLASSIFICATION OF ABSTRACT UNCLASSIFIED	20. LIMITATION OF ABSTRACT UL	

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